

Liquid Chromatograph General Maintenance Protocol

1 Scope

The purpose of this protocol is to provide general guidelines for maintenance of liquid chromatography instruments. This document applies to personnel using the associated instrument(s)/equipment in the following discipline/category of testing: Explosives (chemistry) examinations performed at the Huntsville facility. Refer to the “General Instrument Maintenance Protocol” for overall instrument maintenance guidelines and definitions.

2 Principle

The liquid chromatograph pump is used for sample acquisition, separation of analytes across a column, and to transfer analytes to the detector for identification. The detector may be housed internally, like a conductivity detector, or work externally like a mass spectrometer (MS).

Liquid chromatograph (LC) instruments available for the analysis of evidence are purchased from a variety of manufacturers. All instruments eventually require maintenance, troubleshooting, and repair. Although the user interface and hardware fittings may differ, the overall instrument principles and maintenance are similar. Refer to the “Mass Spectrometry General Maintenance Protocol” for MS system maintenance. Information and procedures concerning performance monitoring of a specific LC can be found in the corresponding instrument’s performance monitoring protocol.

3 Equipment/Materials/Reagents

Any materials (such as columns and filters) and all replacement parts will meet the manufacturer’s specifications and recommendations. Manufacturer’s instrument manuals and specific performance monitoring protocols are generally the best source for this information.

4 Standards and Controls

All standards, solutions, and mobile phases required are specified in the appropriate SOP.

5 Calibration

Any procedures used to verify the integrity of the instrument will be specified in the appropriate SOP.

6 Sampling

Not applicable.

7 Procedures

7.1 Preventative Maintenance

Each type and model of an instrument may have different, specialized components requiring specific preventative maintenance. Suggested step-by-step directions for specific maintenance procedures may be found in the corresponding operations manuals for individual instruments. When performed, all preventative maintenance will be entered into the appropriate log. The following procedures are generic in nature and are included for reference. Daily, monthly, and as needed checks are outlined in the appropriate performance monitoring protocol.

7.1.1 Mobile Phase Filtration

If using high quality High Performance Liquid Chromatography (HPLC) grade reagents, filtering mobile phases is not always necessary. However, filtration is highly recommended, particularly if a buffer is added.

7.1.2 Mobile Phase Degassing

The mobile phase should be degassed prior to entering the LC system in order to remove any dissolved oxygen and carbon dioxide. The LC may be able to provide this capability. If not, try using a helium sparge or a sonicator.

7.1.3 Pump Priming

Perform a 'Wet Prime' prior to connecting the column. Perform a 'Dry Prime' if any of the pump channels are dry.

7.1.4 Pump Cleaning

After use, the entire LC system should be flushed with an organic solvent such as methanol (MeOH) or isopropyl alcohol (IPA). The system should remain in the organic solvent until it is needed again for operation. When buffers are used, the system should be flushed with 100% water for a minimum of ten minutes prior to the organic solvent flush.

7.1.5 Column Maintenance

- a. Prior to use, the column must be equilibrated.

- b. After use, the column should be flushed and stored under appropriate conditions.
- c. Guard columns should be used to increase column lifetime.

7.2 Corrective Maintenance

Each type and model of LC may have different, specialized components requiring specific corrective maintenance. Suggested step-by-step directions for specific procedures may be found in the corresponding manufacturer's instrument manuals for individual instruments. When performed, relevant corrective maintenance will be entered into the appropriate log. The following procedures are more complex options that may be pursued when troubleshooting the LC. The following steps are generic in nature and are included for reference.

7.2.1 Check Valves

The check valves can become dirty and degrade performance over time. They will be replaced as needed. To replace the check valves:

- a. Submerge the check valves in mobile phase or MeOH for several minutes prior to installation.
- b. Loosen and remove the tubing that connects to the check valve housing.
- c. Loosen and remove the check valve housing.
- d. Remove the check valve, noting the orientation in the housing.
- e. Place the new check valve in the housing using the proper orientation.
- f. Place the check valve housing back in the pump. Warning: Do not over-tighten check valve housing.
- g. Reconnect and tighten the tubing that connects to the check valve housing.
- h. Repeat for each check valve.

7.2.2 Pump Seals

The pump seals can deteriorate over time, causing inconsistent or poor pump operation. They should be replaced as needed. To replace pump seals:

- a. Remove all tubing and fittings connected to the pump head.

- b. Loosen the pump head retaining bolts evenly until the head can be removed from the pump.
- c. Remove the rod and fittings to expose the pump seal.
- d. Remove the old pump seal and clear any debris. The area can be washed with methanol or IPA if necessary.
- e. Replace the rod and fittings.
- f. Place the pump head back in the pump and tighten the retaining bolts.
- g. Reconnect all tubing and fittings to the pump seal.
- h. Repeat for each pump head.

8 Instrumental Conditions

Refer to the appropriate procedures outlined in section 7, manufacturer's instrument manuals, or the instrument's performance monitoring protocol for specific instrumental conditions to be used during maintenance procedures.

9 Decision Criteria

Every performance monitoring protocol will have specific decision criteria to determine if the instrument is operating properly. If these should fail, the operator should refer to the 'Corrective Maintenance' section of this protocol.

10 Calculations

Not applicable.

11 Measurement Uncertainty

Not applicable.

12 Limitations

Only properly trained personnel will perform duties involved in the operation, maintenance, or troubleshooting of any instrument. Instrument-specific limitations will be specified in the appropriate SOP.

13 Safety

Take standard precautions for the handling of all chemicals, reagents, and standards. Refer to the *FBI Laboratory Safety Manual* for the proper handling and disposal of all chemicals. Personal protective equipment should be used when handling any chemical and when performing any type of analysis. Many instrument components are held at temperatures of 250°C and higher. Precautions should be taken to prevent the contact of skin with heated surfaces and areas.

14 References

Instrument Operations Group SOP Manual.

Manufacturer's Instrument Manuals for the specific models and accessories used.

"General Instrument Maintenance Protocol" (IOG 001) *Instrument Operations Group SOP Manual.*

"Mass Spectrometer General Maintenance Protocol" (IOG 004) *Instrument Operations Group Subunit SOP Manual.*

FBI Laboratory Safety Manual.

FBI Laboratory Quality Assurance Manual.

FBI Laboratory Operations Manual.

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0	10/04/18	New document which specifies instrument protocol for the Huntsville facility.

Approval

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Scientific Analysis
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TL Approval

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